Shear Flow Induced Crystallization of Isotactic Polypropylene - WAXD

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Beamline(s): **X3A2**

Introduction: The *in situ* WAXD experiments provide a way of determining the nature of the crystal structure development immediately after the application of deformation. It is generally known that the oriented structures form under the influence of shear flow, also the formation of β-form crystals in i-PP after shear has been observed, using light and electron microscopy, by several researchers in the field. The aim of this research project is to study the nature of the crystalline phase in isotactic polypropylene (i-PP) after the cessation of imposed shear flow-field using *in situ* synchrotron WAXD (wide-angle x-ray diffraction).

Methods and Materials: A Linkam CSS-450 high temperature shearing stage modified for in-situ wide-angle x-ray diffraction studies was used to precisely control shear-field and thermal history of the polymer (i-PP) samples. Synchrotron x-ray measurements were carried at the X3A2 beamline in NSLS; a 2D MAR CCD detector was used for the detection of 2D scattering patterns.

Results: Figure 1 shows the WAXD patterns of the i-PP melt at 140 0C after imposition of a step shear-flow field (shear rate 102 s -1, strain 1428%). The WAXD patterns show rapid (few seconds) development of orientated crystals in the melt. It is well known that under quiescent conditions i-PP crystallizes in the most thermodynamically stable α-phase from the melt. After application of the shear field, formation and growth of the β-phase was clearly observed from the x-ray diffraction patterns. The strong β-phase reflection, (300) is seen in addition to the (110), (040), (130), (111 & -131) reflections of α-phase. From the WAXD patterns the total crystallinity as well as the contribution of the β-phase to the crystalline phase was calculated. The total crystallinity rises to a value of about 30% in about 250 s and reaches a plateau of around 40% in less than 500 s. The β-phase grows rapidly and its contribution to the total crystallinity is about 60% after 500 s. The β-phase reflection (300) also shows that it is primarily unoriented. In our SAXS experiments we observed that the crystallization kinetics of the unoriented crystals increase by two orders of magnitude. This observation can be attributed to the β-phase growth observed in the WAXD data. The WAXD patterns show that the α-phase reflections appear first, after the cessation of shear, and the β-phase growth is seen soon afterwards. We speculate that the surface of the oriented α-form crystals act as nucleation sites for growth of the unoriented β-form crystals.

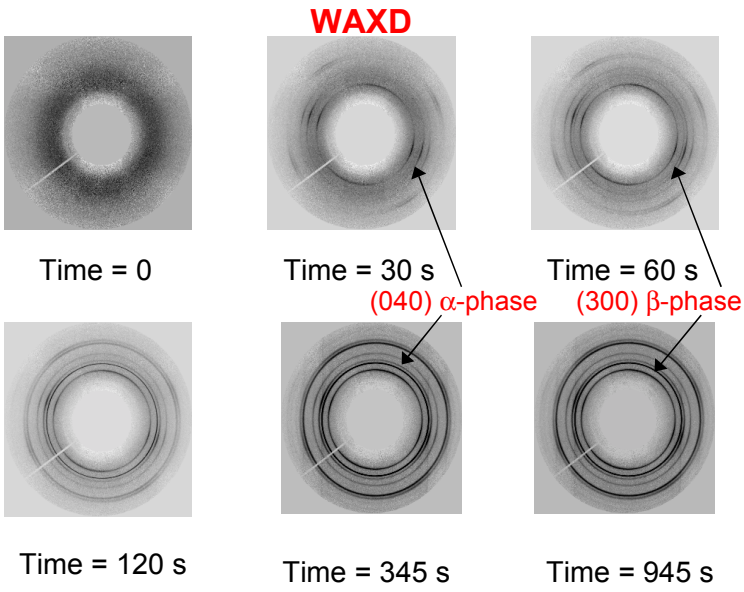

Figure 1. SAXS pattern of I-PP at 140 0C, after step shear (rate = 102 s -1, strain = 1428%); image obtained 900 s after cessation of shear.

Conclusions: The WAXD studies show growth of the β-form crystals along with the α-form in the i-PP melt at 140 ^{0}C after cessation of the applied shear field. The amount of β-phase in the total crystalline phase is dependent on the shear rate as well as the temperature. The WAXD patterns also show that the β-phase crystals are unoriented. The increase in crystallization kinetics of unoriented crystallites is related to the formation and growth of the β-form crystals.

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